FORM PTO-1390 U.S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 50395-084

U.S. APPLIC. NO. (if known, see 37 CFR 1.5): 09/763025

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/JP99/04609	August 26, 1999	August 31, 1998 and January 8, 1999

TITLE OF INVENTION

METHOD OF PRODUCING GLASS ARTICLE AND GLASS BASE MATERIAL FOR OPTICAL FIBER

APPLICANT(S) FOR DO/EO/US

Tomohiro ISHIHARA, Tatsuhiko SAITOH, and Yuichi OHGA

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendment has NOT expired.
   d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information.

    1. International Search Report by Japanese Patent Office
    2. International Preliminary Examination Report
    3. Cover Sheet of Published International Application

U.S. APPLIC. NO. (if known, see 37 CFR 1.50)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/763025	PCT/JP99/04609	50395-084

	CALCULATIONS	PTO USE ONLY
17. ☒ The following fees are submitted:		
Basic National Fee (37 CFR 1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO $860.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) $690.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) $710.00		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO $1,000.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) $100.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 860.00	
Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

Claims	Number Filed	Number Extra	Rate		
Total Claims	8 -20 =	0	x $18.00	$	
Independent Claims	2 -3 =	0	x $80.00	$	
Multiple dependent claim(s) (if applicable)			+ $270.00	$	
TOTAL OF ABOVE CALCULATIONS =				$ 860.00	
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				$	
TOTAL NATIONAL FEE =				$ 860.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property +				$ 40.00	
TOTAL FEES ENCLOSED =				$ 900.00	
				Amount to be: refunded	$
				charged	$

a. ☐ A check in the amount of $ _____ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 500417 in the amount of $ 900.00 to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 500417. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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Stephen A. Becker
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26,527
REGISTRATION NUMBER

February 16, 2001
DATE

BASED ON FORM PTO-1390 (Rev. 5-93)

Docket No.: 50395-084

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	:	
Tomohiro ISHIHARA, et al.	:	
Serial No.:	:	Group Art Unit:
Filed: February 16, 2001	:	Examiner:

For: METHOD OF PRODUCING GLASS ARTICLE AND GLASS BASE MATERIAL FOR OPTICAL FIBER

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, DC 20231

Sir:

Prior to examination of the above-referenced application, please amend the application as follows:

IN THE SPECIFICATION:

Please amend the title to read --METHOD OF PRODUCING GLASS ARTICLE AND OPTICAL FIBER GLASS PREFORM--.

Page 8, line 17, please change ", respectively, reference numerals 8 and 9 denote pipes for" to --, respectively, reference numerals 9 and 8 denote pipes for--.

REMARKS

Entry of this preliminary amendment is respectfully requested.

Respectfully submitted,

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VERIFICATION

The undersigned, of the below address, hereby certifies that he/she well knows both the English and Japanese languages, and that the attached is an accurate English translation of the PCT application filed on August 26, 1999 under No. PCT/JP99/04609.

The undersigned declares further that all statements made herein of his/her own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 11st day of January, 2001

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DESCRIPTION

METHOD OF PRODUCING GLASS ARTICLE AND GLASS BASE MATERIAL FOR OPTICAL FIBER

Technical Field

The present invention relates to a large high-quality glass article, and to a method for manufacturing the same. Specifically, the present invention relates to an optical fiber glass preform which is long and has little variation in outer diameter, and to a method for manufacturing the same.

Background Art

A glass article such as a photo-mask glass preform, optical fiber glass preform, etc., is manufactured by heating at a high temperature to vitrify a soot preform synthesized by a vapor phase synthesis method such as the vapor phase axial deposition (VAD) method, the outside vapor deposition (OVD) method, etc., in a vacuum or in a reduced-pressure atmosphere in a furnace. In order to obtain a high-quality glass article, residual bubbles in the glass preform must be avoided as much as possible and the outer diameter of the preform must be uniform. For these purposes, a method in which the heating and vitrifying step is divided into three distinct steps so that the temperature in each step can be appropriately controlled is proposed (Japanese Unexamined Patent Application Publication No. 6-256035). In this method, a

heating process includes a first heating step of removing the gas remaining in the soot preform, a second heating step of heating at a temperature higher than the heating temperature of the first heating step and lower than a vitrification temperature of the soot preform so as to effect thermal shrinkage, and a third heating step of vitrifying the soot preform at a vitrification temperature. Also, in the second heating step, a heating element for heating the soot preform is divided into several segments in the vertical direction so as to allow independent temperature control in each segment. By setting the temperature of the lower heating segment higher than the temperature in the upper heating segment, variation in the outer diameter of the glass article in the longitudinal direction thereof can be minimized.

Recently, in view of the need for mass production and efficiency in the manufacturing process, a method for manufacturing a high-quality optical fiber preform having a length of 1000 mm or more (which is free of residual bubbles and having little variation in the outer diameter in the longitudinal direction) by using a large soot preform has been desired.

The present inventors have noticed a problem that the variation in outer diameter in the longitudinal direction is increased when an optical fiber glass preform having a length of 1000 mm or more, which is significantly affected by the weight thereof, is treated at a high temperature such as that in the third heating step (1490°C to 1600°C).

The present inventors also noticed a problem in that when the vitrification temperature is lower than 1490°C, and when the amount of

heating time is 1 hour or less, vitrification of the soot preform is difficult and both ends of the soot preform include unsintered portions (imperfectly vitrified portions).

Disclosure of Invention

An object of the present invention is to provide a manufacturing method through which a large high-quality glass article having uniform outer diameter in the longitudinal direction can be manufactured from a large soot preform.

A first aspect of the present invention relates to a method for manufacturing a glass article having a length of 1000 mm or more, comprising a first heating step of inserting a soot preform synthesized by a vapor phase synthesis method into a furnace in a vertical direction and heating to a temperature lower than a vitrification temperature in a vacuum or in a reduced-pressure atmosphere so as to remove gas remaining in the soot preform while effecting thermal shrinkage, and a second heating step of heating the soot preform to a vitrification temperature so as to complete vitrification, wherein, during the second heating step, the temperature at the surface of the soot preform is controlled to be in the range of 1400°C to 1480°C and is maintained thereat for a predetermined period of 70 minutes or more, and wherein a step of cooling the glass article is provided subsequent to the second heating step.

A second aspect of the present invention relates to a method for

manufacturing a glass article according to the first aspect of the present invention, wherein the first heating step has a degassing step of removing the gas to a predetermined vacuum level of 10 Pa or less at a temperature in the range of 1000°C to 1300°C.

A third aspect of the present invention relates to a method for manufacturing a glass article according to the first aspect of the present invention, wherein the first heating step has a degassing step of removing the gas to a predetermined vacuum level of 10 Pa or less at a temperature in the range of 1000°C to 1300°C and a thermal shrinking step of heating to a temperature in the range of 1300°C to 1400°C at a predetermined vacuum level of 10 Pa or less.

A fourth aspect of the present invention relates to a method for manufacturing a glass article according to the first aspect of the present invention, wherein the furnace is provided with a heater having a plurality of segments whose temperatures are independently controllable in the longitudinal direction such that the temperature of the soot preform can be controlled correspondingly in a plurality of parts in the longitudinal direction.

A fifth aspect of the present invention relates to a method for manufacturing a glass article according to the first aspect of the present invention, wherein, during each of the heating steps, the temperature at a furnace tube which isolates the soot preform from a heaters is measured and the temperature in each step is controlled based on the measured values.

A sixth aspect of the present invention relates to a method for

manufacturing a glass article according to the first aspect of the present invention, wherein the soot preform is a composite preform comprising a transparent glass rod and a porous glass portion formed around the glass rod.

A seventh aspect of the present invention relates to a method for manufacturing a glass article according to the first aspect of the present invention, wherein, during the second heating step, the temperature at the surface of the soot preform is gradually or stepwise increased from the upper section toward the lower section.

An eighth aspect of the present invention relates to an optical-fiber glass preform having a length of 1000 mm or more and formed by heating a soot preform which is a composite preform comprising a transparent glass rod and a porous glass portion formed therearound and which has a predetermined outer diameter, so as to vitrify the porous glass portion so that the variation in the outer diameter in the longitudinal direction of the vitrified glass preform is within ±2% relative to the median in the longitudinal direction of the outer diameter.

Brief Description of the Drawings

Figure. 1 is a schematic diagram explaining the structure of a vacuum sintering furnace employed in Examples.

Figure. 2 is a graph showing a variation in outer diameter in the longitudinal direction of the optical fiber preform manufactured in Example 1 and Comparative Example 1.

Figure. 3 is a mimetic diagram showing a state of temperature control in Example 3.

Best Mode for Carrying out the Invention

A soot preform obtained by a vapor phase synthesis method such as the VAD method, or the OVD method, etc., is heated in a vacuum or in a reduced-pressure atmosphere during the first heating step so that the gas remaining in the soot preform is eliminated and the soot preform is thermally shrunk. During this step, it is preferable that the gas be removed to a predetermined vacuum level of 10 Pa or less at a temperature in the range of 1000°C to 1300°C and that a thermal shrinking step be then performed at a predetermined vacuum level of 10 Pa or less at a temperature in the range of 1300°C to 1400°C.

Subsequent to the first heating step, the soot preform is heated to a temperature in the range of 1400°C to 1480°C for 70 minutes or more during the second heating step so as to complete vitrification. By setting the heating temperature to the range of 1400°C to 1480°C, the temperature being lower than a vitrification temperature in the conventional art, elongation of the soot preform by the weight thereof is reduced. In such a case, both end portions of the soot preform, the portions at which the temperature is likely to be lower, may not be vitrified completely; however, by setting the heating time to a predetermined time of 70 minutes or more, the both ends of the soot preform can be completely vitrified. Accordingly, by setting a low vitrification

temperature, variations in the outer diameter of the preform in the longitudinal direction may be within a range of ±2% relative to a median of the outer diameter in the longitudinal direction.

Preferably, the furnace used in the present invention is provided with a heater having a plurality of segments whose temperatures are independently controllable in the longitudinal direction such that the temperature of the soot preform can be controlled correspondingly in a plurality of parts in the longitudinal direction. In this manner, even when a long soot preform is heated, the temperature at a portion which suffers from elongation and the temperature at a portion which suffers less from elongation may be appropriately controlled.

Preferably, when an upright furnace in which the soot preform is inserted in the vertical direction is employed, the temperature at the surface of the soot preform is gradually or stepwise increased from the upper section toward the lower section.

In vitrifying the soot preform, in order to isolate the soot preform from a furnace heater, the soot preform is inserted into a furnace tube comprising carbonaceous material, etc., provided between the soot preform and the heater.

Temperature control during the first and second heating steps is performed by measuring the temperature at the surface of the soot preform by using a thermal sensor such as a radiation pyrometer and controlling the output of the heater based on the measured values. The temperature at the surface of the soot preform is approximately the same as the temperature of

the furnace tube in most cases. Since the temperature at the furnace tube is easier to measure, the output of the heater may be controlled based on the values determined in this way.

EXAMPLES

Hereinbelow, the present invention is more specifically described by way of examples.

(Example 1a)

A soot preform comprising pure silica synthesized by the VAD method was vitrified according to the method of the present invention using a vacuum sintering furnace shown in Fig. 1. In the vacuum sintering furnace shown in Fig. 1, reference numeral 1 denotes a soot preform, reference numeral 2 denotes a vacuum sintering furnace main body, reference numeral 3 denotes a furnace tube, reference numeral 4 denotes a heater, reference numeral 5 denotes an inert gas supply unit, reference numerals 6 and 7 denote flow meters for the inert gas supplied into the furnace tube 3 and the furnace main body 2, respectively, reference numerals 8 and 9 denote pipes for supplying inert gas into the furnace tube 3 and the furnace main body 2, respectively, reference numerals 10 and 11 denote vacuum pumps for reducing the pressure in the furnace, reference numerals 12 and 13 denote pipes for evacuating the furnace main body 2 and the furnace tube 3, respectively, reference numeral 14 denotes a supporting rod for supporting the preform 1, reference numeral 15 denotes an upper cover, reference numeral 16 denotes monitoring hole for

measuring the surface temperature of the preform 1, which extends toward the furnace tube 3, reference numeral 16' denotes monitoring hole for measuring the temperature at the furnace tube 3, reference numeral 17 denotes a pyrometer for measuring the surface temperature of the preform 1, reference numeral 18 denotes a pyrometer for determining a temperature at the furnace tube 3, reference numeral 19 denotes a temperature control device, and reference numeral 20 denotes a traverse mechanism. Means for supplying and evacuating gas may be provided for both the furnace main body 2 and the furnace tube 3 as in the drawing, or for either thereof. In the pipes 8, 9, 12, and 13, valves (not shown in the figure) are provided so that evacuation (pressure reduction) or gas supplying may be carried out by controlling these valves. The pyrometer 18 is connected to the temperature control device 19, though this is not shown in the figure.

The soot preform 1 having an outer diameter of 200 mm, a weight of 30 kg, and an effective length of 1500 mm was synthesized by the VAD method and was used. While maintaining the temperature in the vacuum sintering furnace at 400°C, the soot preform 1 was inserted into the furnace tube 3, the furnace was hermetically sealed with the upper cover 15, and the pressure in the furnace was reduced to 10 Pa. In this state, the surface temperature of the entire soot preform 1 was increased to 1300°C at a rate of 10°C/min and was maintained thereat for 70 minutes so that the gas remaining in the soot preform 1 was thoroughly degassed (degassing step).

The surface temperature of the soot preform 1 was then increased at a

rate of 5°C/min to 1350°C and was maintained thereat for 50 minutes (thermal-shrinking step).

The surface temperature of the soot preform 1 was then increased at a rate of 5°C/min until the surface temperature of the entire soot preform 1 reached 1420°C. The temperature was maintained thereat for 100 minutes to complete vitrification (vitrification step).

Then heating by the heater was discontinued so as to decrease the temperature and to cool the glass article (cooling step), and the product was removed at 600°C.

The size of the obtained glass article was measured and the results showed that the glass article was of superior quality and had little variation in outer diameter, i.e., the outer diameter over the entire effective length of 1400 mm was 90 ±0.5 mm (outer-diameter variation rate was ±0.56%).

(Example 1b)

Instead of the soot preform 1 of Example 1a, a composite preform having the same size as that in Example 1a, the composite preform which had a SiO_2 porous glass layer formed, by using VAD method, around a transparent glass rod having a central portion with a high refractive index doped with Ge and having a pure SiO_2 layer around the central portion, was used. The vitrification was performed as in Example 1a and satisfactory quality was also achieved.

(Example 1c)

A soot preform 1 having an outer diameter of 300 mm, an effective

length of 1500 mm, and a weight of 60 kg was used. As in Example 1a, the degassing step and heating step were performed. The surface temperature of the soot preform 1 was then increased at a rate of 5°C/min until the surface temperature of the entire soot preform 1 reached 1420°C. Then the temperature was maintained thereat for 180 minutes for vitrification.

The obtained glass article was measured and the results showed that the outer diameter was 150 ±1.2 mm (outer-diameter variation rate ±0.6%) over the entire effective length of 1400 mm, achieving high quality.

(Comparative Example 1a)

A soot preform 1 having the same size as that used in Example 1a was vitrified using the same equipment under the conditions below. That is, while maintaining the temperature in the vacuum sintering furnace at 400°C, the soot preform 1 was inserted into the furnace tube 3, the furnace was sealed with the upper cover 15, and the pressure in the furnace was reduced to 10 Pa. In this state, the surface temperature of the entire soot preform 1 was increased to 1300°C at a rate of 10°C/min and was then maintained thereat for 60 minutes so that the gas remaining in the soot preform 1 was thoroughly degassed (degassing step).

The surface temperature of the soot preform 1 was then increased at a rate of 5°C/min to 1350°C and was maintained thereat for 50 minutes (thermal-shrinking step).

The surface temperature of the soot preform 1 was then increased at a rate of 5°C/min until the surface temperature of the entire soot preform 1 was

1500°C. The temperature was maintained for 60 minutes to complete vitrification (vitrification step). Then heating by the heater was discontinued so as to allow the temperature to decrease, and the product was removed at 600°C. The size of the obtained glass article was measured and the results showed a large variation in outer diameter, i.e., the outer diameter over the entire effective length of 1400 mm was 90 ±4.5 mm (outer-diameter variation rate ±5%).

(Comparative Example 1b)

A soot preform 1 having the same size as that used in the Example 1c was used and was vitrified under the same conditions as those in Comparative Example 1a. The size of the obtained glass article was measured and the results showed a large variation in outer diameter in the longitudinal direction, and the outer diameter over the entire effective length of 1550 mm was 150 ±5.0 mm (outer-diameter variation rate ±3%).

The results of Example 1a and Comparative Example 1a are shown in Table 1. The dependence of the outer diameter variation on the longitudinal direction in these examples is shown in Fig. 2. During the second heating step (vitrification step), in order to stabilize the outer diameter, it is apparently important to maintain the temperature in the range of 1400°C to 1480°C (preferably 1400°C to 1440°C) for 70 minutes or more (preferably 100 minutes or more, and more preferably 150 minutes or more) for preventing elongation and for stabilizing the outer diameter.

Although the vacuum level in the furnace is set to 10 Pa in the examples,

, a lower level may be desirable in order to prevent bubbles from remaining in the glass article. The level may be 9 Pa or 8 Pa.

The method of the present invention is effective in reducing the variation in outer diameter, when weight of the glass article is high, particularly when the weight is 50 kg or more.

Table 1: TREATMENT CONDITIONS AND OUTER-DIAMETER VARIATION IN THE OBTAINED GLASS ARTICLE

		Example 1a		Comparative Example 1a	
		Temperature (°C)	Time (min)	Temperature (°C)	Time (min)
First Heating Step	Degassing step	1300	60	1300	60
	Thermal Shrinking Step	1350	50	1350	50
Second Heating Step		1420	100	1500	60
Outer-Diameter Variation (mm)		±0.5		±4.5	

(Example 2a)

A soot preform 1 having the same size as that used in Example 1a was vitrified using the same equipment under the same temperature conditions as those in Example 1a. In this example, however, the temperature control during the vitrification step was performed by determining the temperature at the furnace tube 3 using the pyrometer 18 which measures the temperature at the outer surface of the furnace tube inside of the vacuum sintering furnace. The obtained glass article was measured and the results showed that the glass article had superior quality and a slight variation in outer diameter, i.e., the outer diameter over the entire effective length of 1400 mm was 90 ±0.7 mm (outer-diameter variation rate ±0.78%). These results show that the temperature at the furnace tube may be measured without causing any

problem instead of measuring the temperature at the surface of the soot preform 1. The temperature of the furnace tube is easier to measure.

(Example 2b)

A soot preform having the same size as that used in Example 1c was vitrified using the same equipment under the same temperature conditions as those in Example 1c. The obtained glass article was measured and the results showed that the glass article had superior quality, i.e., the outer diameter over the entire effective length of 1400 mm was 150 ±1.5 mm (outer-diameter variation rate ±1.0%).

(Example 3a)

A soot preform 1 having the same size as that in Example 1a was vitrified as in Example 1a using a vacuum sintering furnace of the same type as that shown in Fig. 3, the furnace having the heaters 4 divided into three units, i.e., an upper heater unit 4-1, a middle heater unit 4-2, and a lower heater unit 4-3 so as to allow independent control therein.

The soot preform 1 having an outer diameter of 200 mm and an effective length of 1560 mm was used. While maintaining the temperature in the vacuum sintering furnace at 400°C, the soot preform 1 was inserted into the furnace tube 3. The furnace was hermetically sealed with the upper cover 15 and the pressure in the furnace was reduced to 10 Pa. In this state, the surface temperature of the entire soot preform 1 was increased to 1300°C at a rate of 10°C/min and was maintained thereat for 60 minutes so as to degas the gas remaining in the soot preform 1 (degassing step).

The surface temperature of the soot preform 1 was then increased to 1350°C at a rate of 10°C/min, and was maintained thereat for 50 minutes (thermal shrinking step).

Next, the surface temperature at the middle of region A of the soot preform 1, the region that is significantly affected by the upper heater unit 4-1, was increased to 1400°C at a rate of 5°C / min, the surface temperature at the middle of region B significantly affected by the middle heater unit 4-2 was increased to 1420°C at a rate of 7°C / min, and the surface temperature at the middle of region C significantly affected by the lower heater unit 4-3 was increased to 1440°C at a rate of 9°C / min. The temperatures were maintained thereat for 100 minutes to completely vitrify the soot preform (vitrification step). The approximate temperature distribution during this step is shown in Fig. 3.

Heating by the heaters was discontinued so as to allow the temperature to decrease and so as to cool the glass article (cooling step), and the product was removed at 600°C.

The outer diameter of the obtained glass article was measured, and the results showed that the glass article had superior quality with little variation in outer diameter, i.e., the outer diameter over the entire effective length of 1405 mm was 90 ±0.1 mm (outer-diameter variation rate ±0.11%).

(Example 3b)

In Example 3a, the control of the surface temperature of the soot preform 1 was performed by determining and controlling the surface

temperature of the furnace tube 3 corresponding to each heater unit. Satisfactory quality was obtained as in Example 3a (the variation in the outer diameter over the entire effective length of 1417 mm was 90 ±0.3 mm (outer-diameter variation rate ±0.33%)).

(Example 3c)

Instead of the soot preform 1 of Example 3a, a composite preform having the same size as that in Example 3a and having a SiO_2 porous glass layer formed, by the VAD method, around a transparent glass rod comprising a central portion doped with Ge and a pure SiO_2 layer formed around the central portion, was used. The vitrification was performed as in Example 3a and satisfactory quality was also achieved.

(Example 3d)

The thermal-shrinking step at a temperature of 1350°C performed in Example 3a was omitted. Satisfactory quality was also obtained.

(Example 3e)

A soot preform 1 having the same size as that in Example 1c was used instead of the soot preform 1 in Example 3a. The vitrification step was performed as in Example 3a except that the holding time was 180 minutes. Satisfactory quality was obtained, i.e., the outer diameter over the entire effective length of 1390 mm was 150 ±0.7 mm (outer-diameter variation rate ±0.46%).

(Example 3f)

In Example 3e, the control of the surface temperature of the soot

preform 1 was performed by determining and controlling the surface temperature of the furnace tube 3 corresponding to each heater unit. Satisfactory quality was obtained as in Example 3a (the variation in the outer diameter over the entire effective length of 1400 mm was 150 ±1.0 mm (outer-diameter variation rate ±0.66%))

(Example 3g)

A soot preform 1 having an outer diameter of 365 mm, an effective length of 1560 mm, and a weight of 80 kg was vitrified by using the same equipment under the same conditions as in Example 3e. The size of the obtained glass article was measured and the results showed that the outer diameter over the entire effective length of 1470 mm was 163 ±1.5 mm (outer-diameter variation ±0.92%).

(Comparative Example 2)

A soot preform 1 having the same size as that used in Example 3g was vitrified using the same equipment as that in Example 3e under the conditions below. That is, while maintaining the temperature in the vacuum sintering furnace at 400°C, the soot preform 1 was inserted into the furnace tube 3, the furnace was hermetically sealed with the upper cover 15, and the pressure in the furnace was reduced to 10 Pa. In this state, the surface temperature of the entire soot preform 1 was increased to 1300°C at a rate of 10°C/min and was maintained thereat for 60 minutes so that the gas remaining in the soot preform 1 was thoroughly degassed (degassing step).

Then the surface temperature of the soot preform 1 was increased to

1350°C at a rate of 5°C/min, and was maintained thereat for 50 minutes (thermal-shrinking step).

Then the surface temperature of the soot preform 1 was increased at a rate of 15°C/min until the surface temperature of the entire soot preform 1 reached 1500°C. The temperature was maintained for 60 minutes to complete vitrification (vitrification step). Then heating by the heaters was discontinued so as to allow the temperature to decrease, and the product was removed at 600°C. The size of the obtained glass article was measured and the results showed that the glass article had a large variation in outer diameter in the longitudinal direction, i.e., the outer diameter over the entire effective length of 1660 mm was 90 ±4.5 mm (outer-diameter variation ±4.43%).

CLAIMS

1. A method for manufacturing glass article having a length of 1000 mm or more, comprising a first heating step of vertically inserting a soot preform synthesized by a vapor phase synthesis method into a furnace and heating to a temperature lower than a vitrification temperature in a vacuum or in a reduced-pressure atmosphere so as to remove the gas remaining in the soot preform while effecting thermal shrinkage, and a second heating step of heating at a vitrification temperature so as to vitrify the soot preform, wherein, during the second heating step, the temperature at the surface of the soot preform is controlled within the range of 1400°C to 1480°C for a predetermined period of 70 minutes or more and wherein a step of cooling the glass article is provided subsequent to the second heating step.

2. A method for manufacturing a glass article according to Claim 1, wherein the first heating step comprises a degassing step of removing the gas to a predetermined vacuum level of 10 Pa or less at a temperature range of 1000°C to 1300°C.

3. A method for manufacturing a glass article according to Claim 1, wherein the first heating step comprises a degassing step of removing the gas to a predetermined vacuum level of 10 Pa or less at a temperature range of 1000°C to 1300°C, and a thermal shrinking step of heating at a temperature range of 1300°C to 1400°C in a predetermined vacuum level of 10 Pa or less.

4. A method for manufacturing a glass article according to Claim 1, wherein the furnace is provided with a heater having a plurality of segments whose

temperatures are independently controllable in the longitudinal direction such that the temperature of the soot preform can controlled correspondingly in a plurality of parts in the longitudinal direction.

5. A method for manufacturing a glass article according to Claim 1, wherein, during each of the heating steps, a temperature at a furnace tube which separates a heater and the soot preform is determined and the temperature in each step is controlled based on the determined temperatures.

6. A method for manufacturing a glass article according to Claim 1, wherein, the soot preform is a composite preform comprising a transparent glass rod and a porous glass portion formed around the glass rod.

7. A method for manufacturing a glass article according to Claim 1, wherein, during the second heating step, the temperature at the surface of the soot preform is gradually or stepwise increased from the upper section toward the lower section.

8. An optical-fiber glass preform having a length of 1000 mm or more and formed by heating a soot preform comprising a transparent glass rod and a porous glass portion formed therearound and having a predetermined outer diameter, so as to vitrify the porous glass portion so that the variation in the outer diameter thereof in the longitudinal direction is within ±2% relative to the median in the longitudinal direction of the outer diameter of the optical glass preform.

ABSTRACT

The present invention relates to a large high-quality glass article and a method for manufacturing the same. Specifically, the present invention relates to an optical-fiber glass preform which is long and has a slight variation in outer diameter. The present invention relates to a method for manufacturing a glass article 1000 mm or longer, the method including a first heating step of heating a soot preform synthesized by vapor phase synthesis method in vacuum or reduced-pressure atmosphere using a furnace at a temperature lower than the vitrification temperature so as to remove gas while performing thermal shrinkage, and a second heating step of heating at the vitrification temperature so as to complete vitrification, wherein during the second heating step, the temperature at the surface of the soot preform is controlled 1400°C to 1480°C for a predetermined time of 70 minutes or more, and wherein a step of cooling a glass article is provided subsequent to the second heating step.

FIG. 1

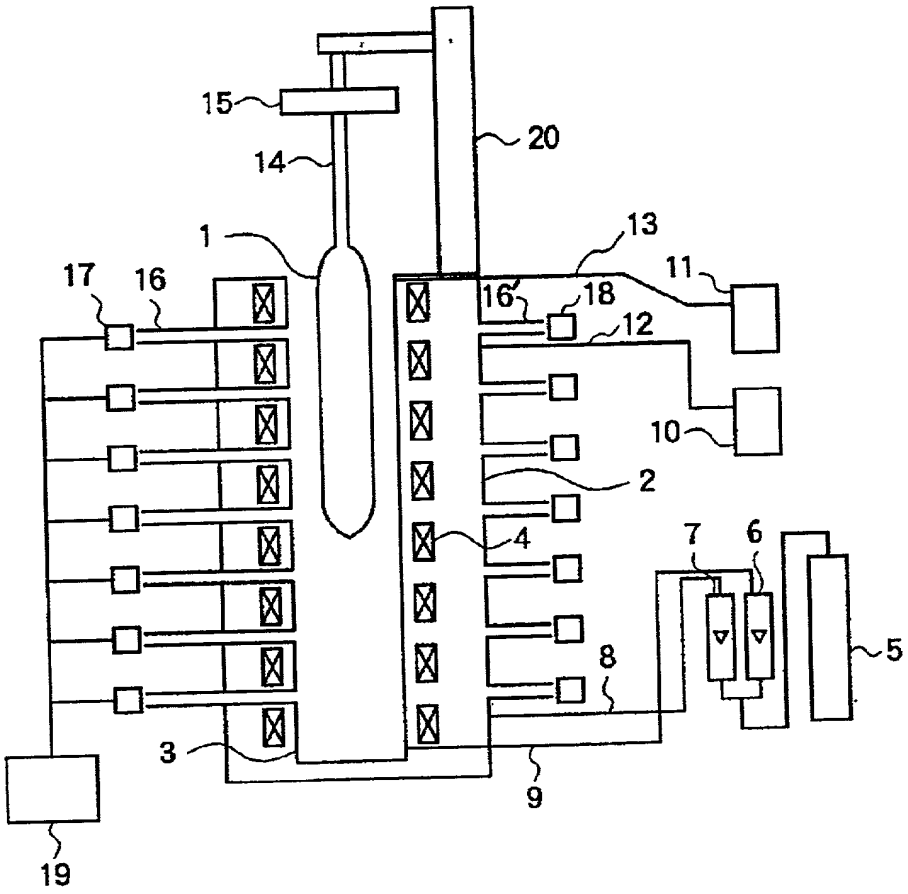


FIG. 2

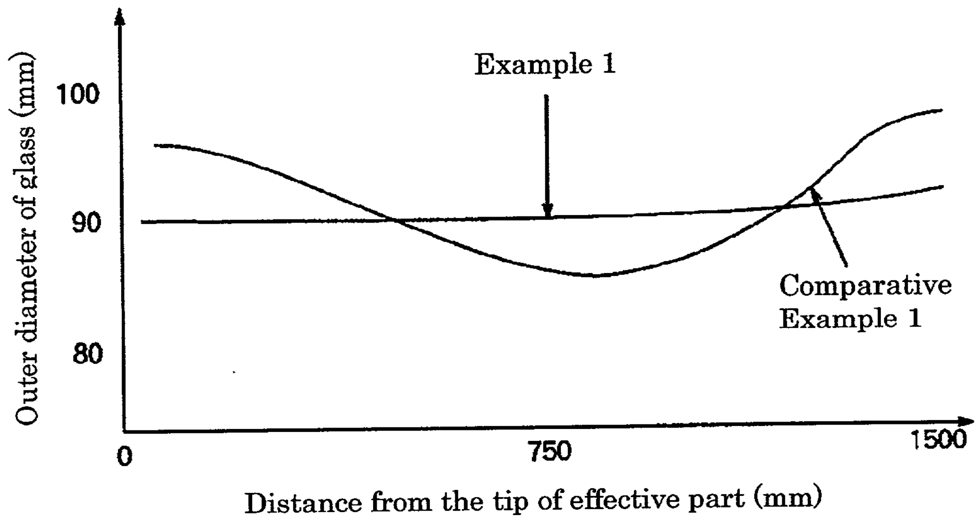
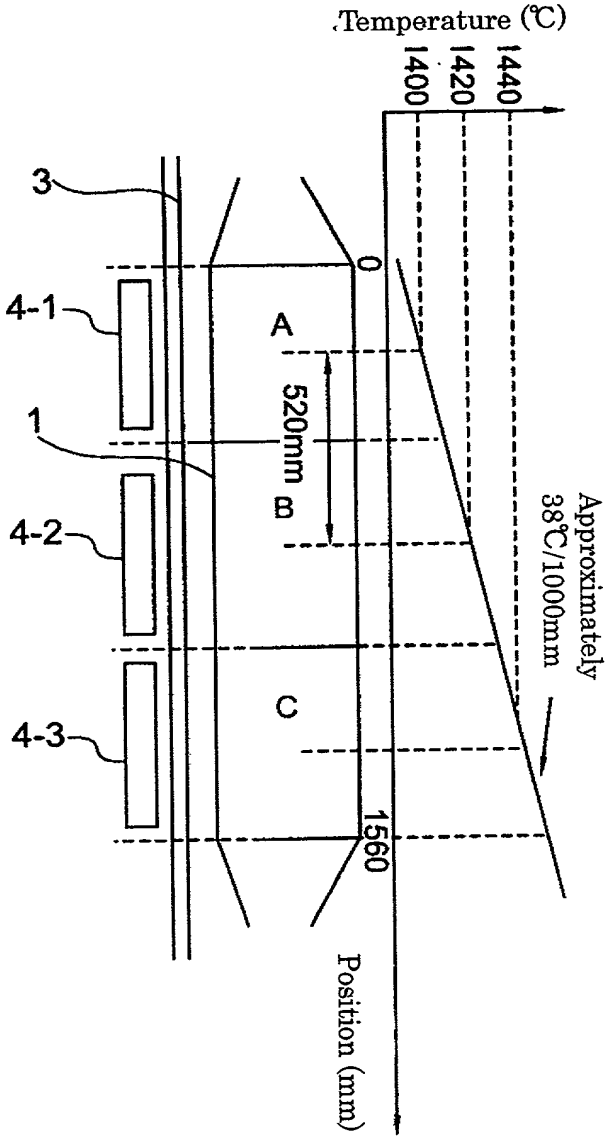


FIG. 3


Temperature (℃)
1400
1420
1440
3
4-1
1
4-2
4-3
0
A
520mm
B
C
1560
Approximately
38℃/1000mm
Position (mm)

1/2 (再)

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT International Application(s)	Attorney's Docket Number

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD OF PRODUCING GLASS ARTICLE AND GLASS BASE MATERIAL FOR OPTICAL FIBER

the specification of which:

☐ is attached hereto.

☐ was filed as United States application Serial No. _____

on _____

and was amended on _____ (if applicable).

☒ was filed as PCT international application Number PCT/JP99/04609

on August 26, 1999

and was amended under PCT Article 19 on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is known to me to be material to patentability in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) or Section 365(b) of any foreign and/or international application(s) for patent or inventor's certificate or Section 365(a) of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (If PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
Japan	10-244809	August 31, 1998	☒Yes ☐ No
Japan	11-002968	January 8, 1999	☒Yes ☐ No

I hereby claim the benefit under 35 USC §119(e) of any United States provisional application(s) listed below.

PRIOR PROVISIONAL APPLICATION(S):

Application Number	Filing Date

(2/2)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s), or §365(c) of any PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application.

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:

U.S. APPLICATIONS		STATUS (Check One)		
U.S. Application Number	U.S. Filing Date	Patented	Pending	Abandoned

PCT APPLICATIONS DESIGNATING THE U.S.					
PCT Application No.	PCT Filing Date	U.S. Serial Numbers Assigned *(if any)*			

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I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signature of Inventor 201:	**Signature of Inventor 202:**	**Signature of Inventor 203:**
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Date Jan. 15. 2001	**Date** Jan. 15. 2001	**Date** Jan. 15. 2001